

CLAIMS:

1. A method of extracting a water-soluble impurity from an aqueous slurry of an arylene sulfide polymer containing the water-soluble impurity, said method comprising:

(a) continuously contacting the polymer containing an impurity directly with sufficient water to produce a slurry containing 3 to 30% by weight of poly(arylene sulfide);

(b) continuously contacting the slurry with a hot heat exchanging medium in indirect heat exchange to raise a temperature of the slurry to a range of 182°C to 216°C;

(c) retaining the slurry at a temperature in a range of 182°C to 216°C for a sufficient time to extract a water-soluble impurity from the aqueous slurry; and

(d) continuously separating the polymer from water.

2. The method according to claim 1, wherein the slurry is heated to a temperature in a range of 182°C to 216°C, and thereafter, the polymer is cooled by indirect contact with a heat exchanging medium prior to separation from water.

Example I

In this Example, a continuous method for extracting a water-soluble impurity, mainly sodium chloride from substantially uncured poly(phenylene sulfide) (PPS) is described.

As shown in FIG. 1, commercially produced Ryton® PPS which had received hot water washing procedures a few times was mixed with hot water in a stirring tank 1 to prepare a slurry having a temperature of about 60°C and containing about 8 to 14% by weight of PPS. The PPS/water slurry was pumped by a high pressure Moyno pump 2 at a rate of 1.5 liters per minute and a pressure of 1379 KPa gauge or greater through a few sections of a pipe having an inner diameter of 1.27 cm and equipped with a series of jackets. A hot heat-conducting oil (sold by Mobiltherm, Mobil oil Company) having a temperature of about 182°C was pumped in a first pipe section 3 having a length of 4.9m through an external jacket in countercurrent with a stream of the PPS/water slurry in an inner tube. The slurry was heated from about 60°C to about 193°C while the heat-conducting oil was cooled to about 82°C. A residence time of the slurry in this pipe section was about 2 seconds.

Then, the slurry was pumped through a few pipe sections 4, whereby, a total residence of about 80 seconds was attained, during which deashing (salt extraction) occurred at about 188°C. Subsequently, the hot slurry was cooled to a final temperature of about 66°C with cold water in a pipe section 5 equipped with a jacket having a length of 2.4 m, collected in a tank 6, and retained therein under a nitrogen pressure of 1379 KPa gauge. Then, a pressure was decreased, the cooled slurry was filtered through 5 square feet Bird Young filter, and optionally, washed with cold water.

CLAIM:

1. A method of continuously discharging a slurry, which comprises introducing a solid polymer particle slurry present in a first region retained at a sufficient pressure for retaining at a liquid phase a diluent comprising at least one kind of hydrocarbon or halogenated hydrocarbon which is a gas at a normal temperature and a normal pressure, into a heating pipe comprising a piping covered with a heating jacket divided into 2 or more, and then, separating and recovering the substantially dried solid polymer particle and the evaporated diluent steam from a second region retained substantially at an atmospheric pressure, said method further comprising controlling an amount of a slurry to be discharged, by the number of heating jackets for introducing a steam and an amount of liquid hydrocarbon and halogenated hydrocarbon which is to be introduced into a heating tube separately and which is a gas at a normal temperature and a normal pressure.

An object of the present invention is to provide a method for separating and recovering a substantially dried solid polymer particle and an evaporated diluent steam by continuously discharging a solid polymer particle slurry while severely controlling an amount of the slurry to be discharged.

As a solid polymer particle which forms a slurry in the

present invention, there are, for example, polyethylene, polypropylene, polybutene-1, polyvinyl chloride and a copolymer thereof. In addition, examples of the diluent include liquefied gases such as propane, propylene, butane, butene and vinyl chloride and a mixture thereof as well as a liquid mixture of them with ethylene or hydrogen. The diluent may further contain around 20% by weight or smaller of a high boiling diluent such as pentane, hexane, heptane, benzene and toluene which are a liquid under a normal temperature and a normal pressure.

A dimension of a heating tube comprising a piping covered with a heating jacket (hereinafter, abbreviated as heating tube simply) in the present invention is different depending on a discharging flow rate of the polymer slurry to be treated, a kind of a diluent, a pressure in a first region and that in a second region, a polymer particle slurry concentration, and the like. However, inner diameters of heating tubes are made to be a relatively large bore diameter and a small bore diameter, heating tubes having a relatively small bore diameter are provided in a first region, and a ratio of the bore diameter is 1.2 to 3-fold. This is effective for widening a control width of an amount of a slurry to be discharged.

A flow rate of a polymer slurry is preferably 3 to 20 m/sec. at an inlet of a heating tube and 14 to 150 m/sec. at an outlet of a heating tube (provided that a slurry is a mixed slurry of a polymer particle and a diluent steam), and a dimension of a heating tube should be determined like that. It is desirable that a pressure in a first region is 10 to 50 kg/cm<sup>2</sup>G, and a

pressure in a second region is 1 to 7 kg/cm<sup>2</sup> G.

In addition, a length and an inner diameter of a heating tube and a size of a heating jacket should be determined so that substantially 100% of a diluent is evaporated in a second region even when operated at a desired maximum slurry discharging rate. Specifically, they can be determined by a known method using the physical property values such as specific heat and evaporation latent heat of a diluent.

When an extent of division of a heating jacket is greater, there is needed a smaller amount of hydrocarbon or halogenated hydrocarbon which are necessary for continuously changing an amount of a slurry to be discharged and is to be introduced into a heating tube. However, practically, a length of a divided jacket is 1 m or more, and particularly 5 m or more.

Examples of liquid hydrocarbon or halogenated hydrocarbon which is a gas at a normal temperature and a normal pressure and which is to be introduced separately, include embodiments of the aforementioned diluent. It is preferable for reutilizing a steam separated and recovered in the second region that same hydrocarbon or halogenated hydrocarbon as that used as a diluent, is used.

By implementing the method of the present invention, it becomes possible to discharge a slurry by controlling an amount of a slurry to be discharged, effectively and severely, being industrially valuable.

The present invention will be further explained by way of Examples.

### Example and Comparative Example

A test of discharging a slurry was carried out using an apparatus shown in FIG. 1.

The apparatus comprises a piping with an inner diameter of 1 inch and a length of 80 m having a heating jacket divided into five sections, and a piping with an inner diameter of 1 1/2 inch and a length of 60 m having a heating jacket. 1.4 kg/cm<sup>3</sup> of a heating steam can be passed through each heating jacket. A heating steam can be or can not be introduced by opening and closing valves 4-1, 4-2, 4-3, 4-4, 4-5 and 7, respectively. A polymer particle and a steam exited from the heating tube are separated by a cyclone 9, a steam is discharged through 11, and a polymer particle enters 10.

A slurry containing 45% (by weight) of a polypropylene particle was discharged from a container maintained at a temperature of 40°C and gauge pressure of 45 kg/cm<sup>2</sup> by fully opening a valve 2. The cyclone 9 is maintained at a gauge pressure of 0.3 kg/cm<sup>2</sup>. The number of heating jackets for introducing a steam was decreased successively to 6, 5, 4, 3 and 2 by starting with the case where 1.4 kg/cm<sup>2</sup> of a steam is passed through all the heating jackets and closing the valves 4-1, 4-2, 4-3 and 4-4 in this order. Then, an amount of a slurry to be discharged was stepwisely changed such as 4.9 ton/hr, 7.0 ton/hr, 8.6 ton/hr, 9.3 ton/hr and 10.3 ton/hr. (Comparative Example) To the contrary, the number of heating jackets was decreased from 6 to 5, and at the same time, liquid propylene was fed at 1.2 ton/hr through a line 12, and an amount of propylene to be feed was

changed, so that an amount of a slurry to be discharged was measured. Then, as an amount of propylene to be fed is decreased, an amount of a slurry to be discharged was continuously decreased, and an amount of slurry to be discharged became 7.0 ton/hr at an amount of to be fed 0. The relationship between an amount of a slurry to be discharged and an amount of fed propylene is shown in FIG. 2.

CLAIM:

1. A method of continuously removing an unreacted vinyl chloride monomer in a polyvinyl chloride suspension or a polyvinyl chloride emulsion to 1000 ppm or lower per resin, said method comprising countercurrent-contacting a polyvinyl chloride suspension or a polyvinyl chloride emulsion from which a majority of an unreacted vinyl chloride monomer has been recovered after completion of a polymerization reaction, with an inert gas at an amount of 1 to 100-fold a volume relative to a flow rate of a fed suspension or emulsion, at a temperature of 75 to 95°C for a time not exceeding 30 minutes in the gas dispersion type state using a packed tower or a tray tower.

According to the method of the present invention, at the aforementioned tower height or step number, for example, the concentration of the unreacted vinyl chloride monomer after treatment can be decreased to 20 ppm by continuously treating the 1000 ppm PVC slurry or the latex. An inner diameter of the tower, that is, a vacant tower cross-sectional area is decided by a feeding amount of the PVC slurry or latex to be treated in the tower. According to the present method using an inert gas as a carrier gas, since the effect of removing an unreacted vinyl chloride monomer is extremely high, a flow rate of the slurry or latex per unit vacant tower cross-sectional area is



approximately 50 cc/min·cm<sup>2</sup>.

#### Example 1

When 3000 kg of deionized water, 2000 kg of vinyl chloride, 0.6 kg of tertiary butyl peroxyvalate and 2 kg of partially saponified polyvinyl alcohol having a saponification degree of 80 mole% were charged into a polymerization machine having an inner volume of 7 m<sup>3</sup>, and a polymerization reaction was carried out at 57°C and at a gauge pressure of 8.8 kg/cm<sup>2</sup>, a pressure was decreased to a gauge pressure of 5 kg/cm<sup>2</sup> after 11 hours. Then, before a gauge pressure at 57°C reached 0 kg/cm<sup>2</sup>, the unreacted vinyl chloride monomer was recovered, and further, degassing was performed at 70°C for 2 hours under reduced pressure using a vacuum pump having an evacuating rate of 5 m<sup>3</sup>/min. Thereafter, the concentration of the remaining unreacted vinyl chloride monomer in the PVC slurry was measured and found to be 950 ppm when converted into per resin.

The PVC slurry from which a majority of the unreacted vinyl chloride monomer had been removed by degassing under reduced pressure in advance, was subjected to continuous unreacted monomer removal by the apparatus system shown in FIG. 1.

That is, the PVC slurry was discharged through a bottom of a polymerization machine 1, this was transferred to a receiver 3 having a volume of 1 m<sup>3</sup> via a pump 2, and thereupon, a steam was blown therein through a steam blowing tube 4 so that a temperature of a slurry was maintained at 70°C. This slurry

was supplied continuously at a flow rate of 16 l/min from a tower top of a packed tower 5 packed with 1 inch Telalette packing and having an inner diameter of 20 cm and a tower height of 800 cm. On the other hand, nitrogen was blown therein at a flow rate of 64 l/min by a nitrogen introducing tube 6 from the tower bottom of the packed tower 5. During this, a steam was blown therein by a steam introducing tube 7 from the tower bottom of the packed tower 5 so that a temperature of the slurry in the packed tower 5 was maintained at 90°C. The packed tower 5 was filled with the slurry to a Telalette packing filling position as described above, and was operated in a gas dispersion manner. The slurry treated in the packed tower 5 was continuously discharged from a tower bottom of the packed tower 5, transferred from a tower top of a heat exchanger 8 to the heat exchanger, where the slurry was indirectly cooled to 60°C with a cooling water which is to be supplied to the heat exchanger 8 by a cooling water introducing tube 9 and to be discharged from the heat exchanger 8 by a cooling water introducing tube 10.